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Test Procedure for

**PREPARATION OF FLY ASH SAMPLES FOR  
CHEMICAL ANALYSIS BY X-RAY FLUORESCENCE**



**TxDOT Designation: Tex-318-D**

**Effective Date: June 2012**

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**1. SCOPE**

- 1.1 This method covers the preparation of Class F and Class C fly ash for chemical analysis by X-ray fluorescence (XRF) using a fused pellet.
  - 1.2 The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.
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**2. APPARATUS**

- 2.1 *Analytical balance, Class A in accordance with Tex-901-K.*
  - 2.2 *Beaker, 100 mL.*
  - 2.3 *Watch glass, large enough to cover the beaker.*
  - 2.4 *Fusion instrument.*
  - 2.5 *Suction cup*
  - 2.6 *Platinum crucibles, 5% Au and 95% Pt.*

**Note 1**—Each fusion instrument will require a different size and shape crucible. Follow manufacturer's recommendations.
  - 2.7 *Platinum molds, 5% Au and 95% Pt.*

**Note 2**—Each fusion instrument will require a different size and shape mold. Follow manufacturer's recommendations.
  - 2.8 *Latex gloves.*
  - 2.9 *Adhesive labels, approximately 1/2 × 1/2 in.*
  - 2.10 *Spatula, sized appropriately to transfer the material into the crucibles.*
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- 2.11        *Desiccator.*
  - 2.12        *Muffle furnace, able to heat to 1000°C.*
  - 2.13        *Crucibles and covers, able to withstand 1000°C.*
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### 3.            **MATERIALS**

- 3.1        *Lithium Borate-Lithium Bromide, 66.67%  $\text{Li}_2\text{B}_7\text{O}_4$  / 32.83%  $\text{LiBO}_2$  / 0.50%  $\text{LiBr}$ .  
(Reagent must be “Pure Grade.”)*

**Note 3**—This is referred to as *flux* in the procedure.

- 3.2        *Fly ash samples.*
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### 4.            **PROCEDURE**

**Note 4**—Prior to fusing the sample, it is extremely important to take the utmost care not to contaminate the samples. It is important that any surface that will be presented to the XRF instrument for analysis be kept flat and contaminant free. The procedure requires that the preparer touch the sample. When doing so, be careful not to touch the fused bead on the surface that is to be analyzed.

- 4.1        Wash hands and put on gloves. Do not touch anything that may contaminate the samples, (face, hair, etc.)
  - 4.2        Perform Moisture Content and Loss on Ignition (LOI) tests with the fly ash samples in accordance with ASTM C 311.
  - 4.3        Place molds in the corresponding holders in the fusion instrument. Be sure that the molds fit evenly in the holders.  
**Note 5**—Do not touch the inside surface of the molds.
  - 4.4        Place a clean beaker on the scale and tare the scale.
  - 4.5        Pour approximately 24.0 g of flux into the beaker. Take the beaker off the scale and cover with the watch glass.  
**Note 6**—Never add anything to the flux bottle, including spatulas and excess chemical. Transfer any excess flux from the beaker into a plastic bag to clean the crucibles when testing is complete.
  - 4.6        Remove a crucible from its container and place it on the scale. Tare the scale.  
**Note 7**—Do not touch the inside of the crucible.
  - 4.7        Weigh  $6.5 \pm 0.0005$  g of flux into the crucible from the beaker. Cover the beaker with the watch glass.
  - 4.8        Tare the scale again.
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- 4.9 Weigh  $0.5 \pm 0.0005$  g of ignited fly ash into the crucible.  
**Note 8**—The amount of sample and flux used is dependent on the size of disk that will fit in the holder of the spectrometer used. Use the appropriate amount of sample and flux to produce disks approximately 2 mm thick.  
**Note 9**—Crush any clumps in the fly ash before weighing the sample.
- 4.10 Carefully, with the spatula, mix the flux and the sample together.  
**Note 10**—Carefully, using a flat spatula and only if necessary, tap the outside of the crucible to remove the sample from the inside walls and double check that the weight was not altered.
- 4.11 On an adhesive label, record the sample identification number and the crucible number containing the sample, e.g., D0530150-2A.
- 4.12 Place crucible in fusion instrument.
- 4.13 Repeat Sections 4.6–4.12 for the remaining crucibles.
- 4.14 Operate fusion instrument according to manufacturer's recommendations.
- 4.15 Once the material is fused, it should have a disk shape and be transparent. Check the disk for imperfections, such as spots or cracks. If the disk does not show any imperfection, proceed to Section 4.16. If there are imperfections, discard the disk and re-fuse the sample.
- 4.16 With the sample still in the mold, place the adhesive label with the lab number on the top surface.
- 4.17 Remove each sample from its mold with the suction cup and place the disk on the desiccator tray. Take care not to touch the surface of the sample that was in contact with the mold or place the disk on a surface other than the desiccator tray.
- 4.18 Repeat Sections 4.15–4.17 for all remaining samples.  
**Note 9**—If necessary, clean crucibles by running a fusion using approximately 7 g of flux. Never touch the inside of the crucibles with your fingers, scratch the crucibles, or use any metallic instrument to clean the crucibles.
- 4.19 Carefully remove each mold without touching the inside surface. Place each mold into its corresponding plastic container.
- 4.20 If cleaning is not necessary, store crucibles and molds according to any standard operating procedure in effect for safety and security.
- 4.21 Switch off the main gas supply and turn off the instrument.